DEPARTMENT OF TRANSPORTATION
ENGINEERING SERVICE CENTER
Transportation Laboratory
5900 Folsom Blvd.
Sacramento, California 95819-4612

METHOD OF DETERMINING ASPHALT CONTENT OF BITUMINOUS MIXTURES BY VACUUM EXTRACTION

CAUTION: Prior to handling test materials, performing equipment setups, and/or conducting this method, testers are required to read "**SAFETY AND HEALTH"** in Section I of this method. It is the responsibility of the user of this method to consult and use departmental safety and health practices and determine the applicability of regulatory limitations before any testing is performed.

A. SCOPE

The procedure for removing all the asphalt from a bituminous mixture using a cold solvent and vacuum in order to obtain the asphalt content of the mix is described in this method.

B. APPARATUS

1. Extraction assembly consisting of (Figure 1):
   a. Funnel ring (sample container).
   b. Sludge container.
   c. Filter support.
2. Balance: 4.5 kg capacity, 0.1 g sensitivity.
3. Ultrasonic bath (frequency 50,000 Hz) (Figure 1).
4. Microwave oven: approximate interior dimension of 300 by 400 by 200 mm.
5. Exhaust hood or fan, or portable cart with exhaust fan (Figure 1).
6. Vacuum pump, minimum capacity: pump, 670 mm Hg, system 115 L/min (Figure 2).
7. Curing pans: approximately 300 by 200 by 30 mm, one Pyrex dish.
8. Gloves: heat resistant.
9. Gloves: rubber.
10. Mechanical mixing machine - optional.
11. Small pointed trowels.
12. Beakers: 1000 mL.
13. Plastic squeeze bottle for washing.

C. MATERIALS

1. Filter paper (Grade 617, 33 cm VWR28313-181 or equivalent).
2. Solvent (Trichloroethane, Technical Grade 1,1,1, inhibited).
3. Diatomaceous earth (Celatom MW27).
4. Vegetable oil (Wesson oil or equivalent–Wesson to be used in case of dispute).
5. Storage drum or container for used solvent.
6. Tap water for ultrasonic bath.

D. PREPARATION OF SAMPLES

1. Obtain approximately 8 kg of material (field sample or batch).

2. Split material to approximately 4 kg using the sample splitter indicated in Figure 1 of California Test 304, or by use of a riffle splitter conforming to the size indicated in California Test 201 or California Test 125.

   NOTE: In lieu of splitting, the sample may be reduced to testing size by quartering using the procedures in California Test 125.

3. Split the sample again to obtain a test mass of approximately 2 kg.

   a. Use one part to determine the asphalt content. (In some instances, such as when testing mixes containing recycled AC, 1 kg portions may have to be used to avoid plugging the filter. This will require separating the extraction sample into two parts.)

   b. Split or quarter the other 2 kg portion to obtain approximately 500 g for the moisture determination. Save the remaining amount for a check sample.

      NOTE: Mixtures containing emulsion or cut-back asphalt must be pre-dried to a constant mass prior to extraction.

   c. Heat or cool the sample to a temperature between 80˚C and 125˚C prior to testing. The solvent will boil at 77°C. (Splitting mixtures after sampling usually drops the temperature 14 ± 3°C.)

E. EXTRACTION CALIBRATION CURVE (Complete only if job aggregate is available)

1. Prepare six 2 kg aggregate samples using the aggregate and gradation proposed for the project (1 kg samples may have to be used; see D.3.a., above).

2. Bring aggregate and asphalt to 150 ± 6˚C.

3. Prepare triplicate test specimens with asphalt contents at least 1.0 % above and 1.0 % below the Optimum Bitumen Content (OBC). Mix them for 3 min ± 10 s (hand mix or machine mix).

4. After mixing, place in curing pan and cure at room temperature for 15 to 20 h.

   NOTE: Omit this step if control samples are to be extracted the day they are mixed.

5. Extract binder as per Section F.

6. Dry all emulsion mixes for 5 h (minimum) in a 150°C oven prior to testing, or one hour in a microwave oven. Cool them to room temperature, and test as indicated in Section F.

F. TEST PROCEDURES

1. Determine the moisture content of the sample using:

   a. California Test 370 (microwave oven) or

   b. California Test 310 (for moisture content only).

2. Set up extraction equipment (requires water, electricity, solvent, vegetable oil, and diatomaceous earth).

3. Place one tared filter paper on filter support (Figure 3).

4. Place funnel ring over filter and secure it tightly with wing nuts tightened by hand (Figure 4).

5. Weigh 30 g of diatomaceous earth and mix it with approximately 500 mL of

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A slurry consisting of diatomaceous earth and either water or trichloroethane will work satisfactorily. However, since the solvent used for extraction is usually reclaimed, it is preferable to use a slurry for the filter consisting of trichloroethane and diatomaceous earth.

trichloroethane (60 g may be required to avoid plugging the filter when testing a mix that contains recycled AC).

6. Turn the vacuum on and pour the mixture into the funnel ring. Vacuum it until the earth appears dry.

7. Place sample in tared Pyrex dish, weigh dish and sample, and record mass. (For samples that have been cooled, bring temperature to 110 ± 10°C prior to testing.)

8. Add 100 mL of vegetable oil.

9. Stir mixture for 30 s and place in microwave oven for 5 min.

10. Turn on exhaust fan and ultrasonic bath.

11. Remove sample from microwave oven, and place it in the ultrasonic bath sample container. (Note: The water in the ultrasonic bath should be just deep enough so as not to overflow when sample container is in place).

12. Stir for approximately 1 min.

13. Pour in 1000 mL of solvent. (Note: Solution may boil for first few seconds.)

14. Stir constantly for 4 or 5 min with ultrasound on.

15. Turn on vacuum pump and place a small pan on the filter so the diatomaceous earth is not disturbed when the liquid is poured. Pour dirty solvent from sample slowly into a small pan. If the filter becomes plugged, test subsequent samples by pouring the dirty solvent from each wash into separate containers, then pouring the dirty solvent from the fourth wash into the pan followed by the solvent from wash 3, then 2, and then the solvent from the first wash. Use the maximum vacuum available.

16. Return the sample container to the sonic bath and repeat steps 11 (excluding microwave oven) through 15 until a total of 4 washes have been completed.

17. When repeating steps 13 through 15, do not pour additional liquid into the extractor until the previous solvent has been vacuumed through the filter. If extraction takes longer than the 4 to 5 min, the diatomaceous earth surface may be scratched lightly. To expedite filtering of subsequent samples from the same source, the use of additional diatomaceous earth may be beneficial (try using 60 g).

18. Pour the entire mixture onto the filter. Use wash bottle containing solvent to cleanse the ultrasonic bath sample container.

19. Pour 700 mL of solvent into the funnel ring, stir the sample for approximately 2 min, and vacuum it. Repeat until three washes have been completed.

20. After the third wash, let the vacuum run for an additional 2 or 3 min, or until the aggregate appears dry. Then scrape the material away from the edge of the funnel ring, turn off the vacuum, and place the filter paper and sample into the tared Pyrex dish. Turn off exhaust fan.

21. Place Pyrex dish in microwave oven, and dry sample to constant mass (approximately 20 min).

22. Record the extracted aggregate mass, correct for moisture, and determine the asphalt content. (If moisture content is not immediately known, a previously known moisture content may be used until moisture content has been established.)

G. CALCULATIONS

1. Calculate the percentage of bitumen using the following formula (Worksheet TL-307 may be used, Figure 5):

   a. % moisture = [(mass of moisture)/ (dry mix mass)] x 100

Where:

Dry mix mass = mass of asphalt and aggregate after drying

b. % bitumen = [(mass of asphalt removed)/(dry mass of extracted aggregate)] x 100

Where:

mass of asphalt removed = [(total mass) - (dry mass of extracted aggregate) - (mass of moisture)].

2. Establish a calibration curve to adjust the extracted data.

   a. For the calibration curve, average the results of three test specimens for each percent bitumen. Plot the extracted asphalt content versus the actual asphalt content on Form No. TL-309 and connect these points with a straight line (Figure 6).

   b. Use this curve to correct for bitumen content prior to reporting. Correct the test results of extracted samples as follows:

      Locate the extracted asphalt content on the ordinate, proceed horizontally to the calibration curve. At the intersection, drop vertically to obtain actual asphalt content indicated on abscissa.

H. REPORTING TEST RESULTS

1. Report the bitumen content of field control samples and research samples after the correction using the calibration curve (G.2.a). Use the letter "C" and the calibration curve number (encircled) with each corrected test result.

   Example: 4.8 % C ③

2. Adjust the calculated bitumen content (G.1) on cores, recycled AC, or research samples on old pavement by adding 0.2 % to approximate the unrecovered asphalt. Use the letter C with each test result to indicate correction has been applied.

   Example: 4.5 % C

3. Perform the sieve analysis according to California Test 202, Appendix A, and record the data in percent passing each sieve based on the dry mass of the extracted aggregate.

4. Report on Form TL-302. Identify California Test 362 on report.

I. SAFETY AND HEALTH

Care should be taken when handling solvents. Use rubber gloves, eye protection, and work in a ventilated or hooded area to avoid breathing concentrated fumes, or use the portable extraction cart designed for this test (Figure 1).

Suitable gloves should be worn when handling hot material.

Prior to handling, testing or disposing of any waste materials, testers are required to read: Part A (Section 5.0), Part B (Sections: 5.0, 6.0 and 10.0) and Part C (Section 1.0) of Caltrans Laboratory Safety Manual. Users of this method do so at their own risk.

References:
California Tests 125, 201, 202, 304, 310, and 370

End of Text
(California Test 362 contains 8 Pages)

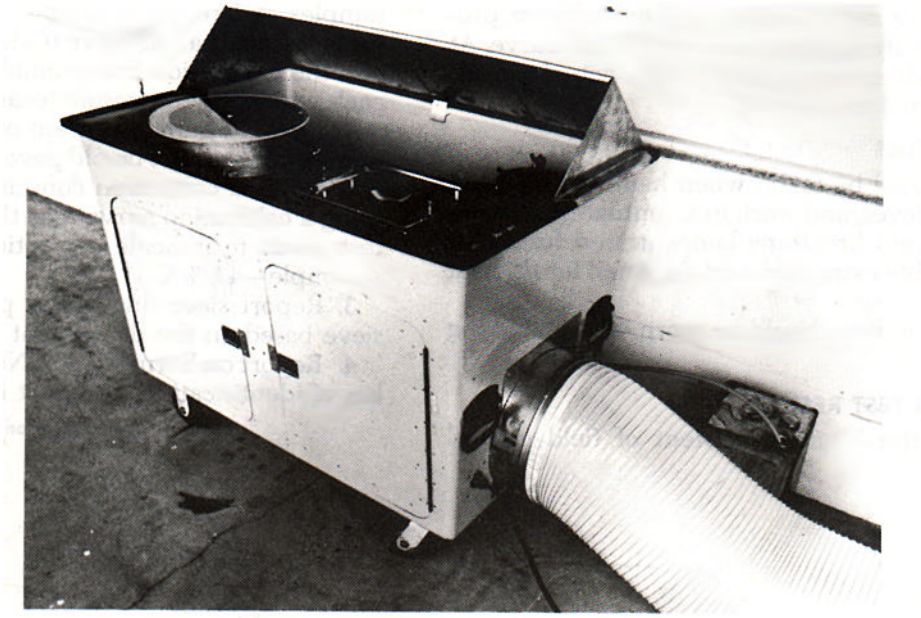

Figure 1
Ultrasonic Bath, A Commercial Extractor and Vacuum Pump

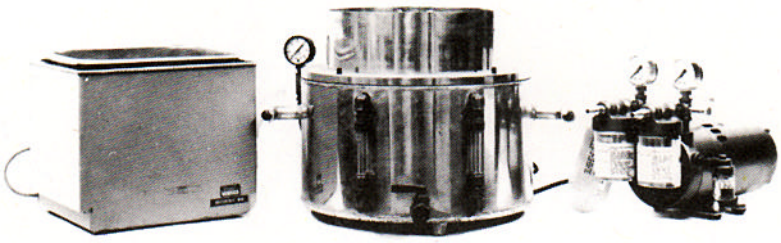

Figure 2
Portable Cart With Exhaust Fan

Figure 3
Filter Paper Being Placed on Support

Figure 4
Funnel Ring Being Placed on Filter Support

California Department of Transportation
Transportation Laboratory
Pavement Testing and Standards Branch
TL-307 (Rev. 11/95)

A.C. VACUUM EXTRACTION

Contract No. _____ **Res. Engineer** _____

Date Sampled _____ **Plant** _____

Before Extraction			
A	Total Mass		
B	Pan Tare		
C	Sample Mass		
After Extraction			
D	Total Extracted Mass		
E	Diatomaceous Earth		
F	Filter Paper		
G	Pan Tare		
H	Sample Net Mass = D-E-F-G		
I*	Moisture (Gram)		
J	(C) –(H) =		
Extracted Asphalt Content (J) –(I) = (H)		%	%
Design Asphalt Content		%	%
Asphalt Source		Grade of Asphalt	

Sieve Analysis				
Size			Spec.	Moisture Calculations
25.0 mm				Mass to Deduct for Moisture
19.0 mm				$*I = \frac{C}{K} \times M$
12.5 mm				*Where*:
9.5 mm				C = Mass of the extraction sample
6.35 mm				K = Mass of the moisture sample
4.75 mm				M = Moisture Loss in grams
2.35 mm				(1 gram = 1 mL)
1.18 mm				Determined by
600 µm				California Test 370 or
75 µm				California Test 310

Remarks:

Figure 5

EXTRATION CALIBRATION CURVE NO ___3___

TEST METHOD___CA 362___

DISTRICT____10____ CONTRACT NUMBER____123456____

AGGREGATE SOURCE_____Browns Pt.____ AGGREGATE Km VALUE____1.4____

ASPHALT GRADE____AR-4000___ ASPHALT SOURCE____Chevron-Oakland____

DATE July 11, 1984

GRADATION OF AGGREGATE USED								
SIEVE SIZE	25.0 mm	19.0 mm	12.5 mm	9.5 mm	4.75 mm	2.36 mm	600 μm	75 μm
% PASSING	100	98	85	72	52	35	18	4

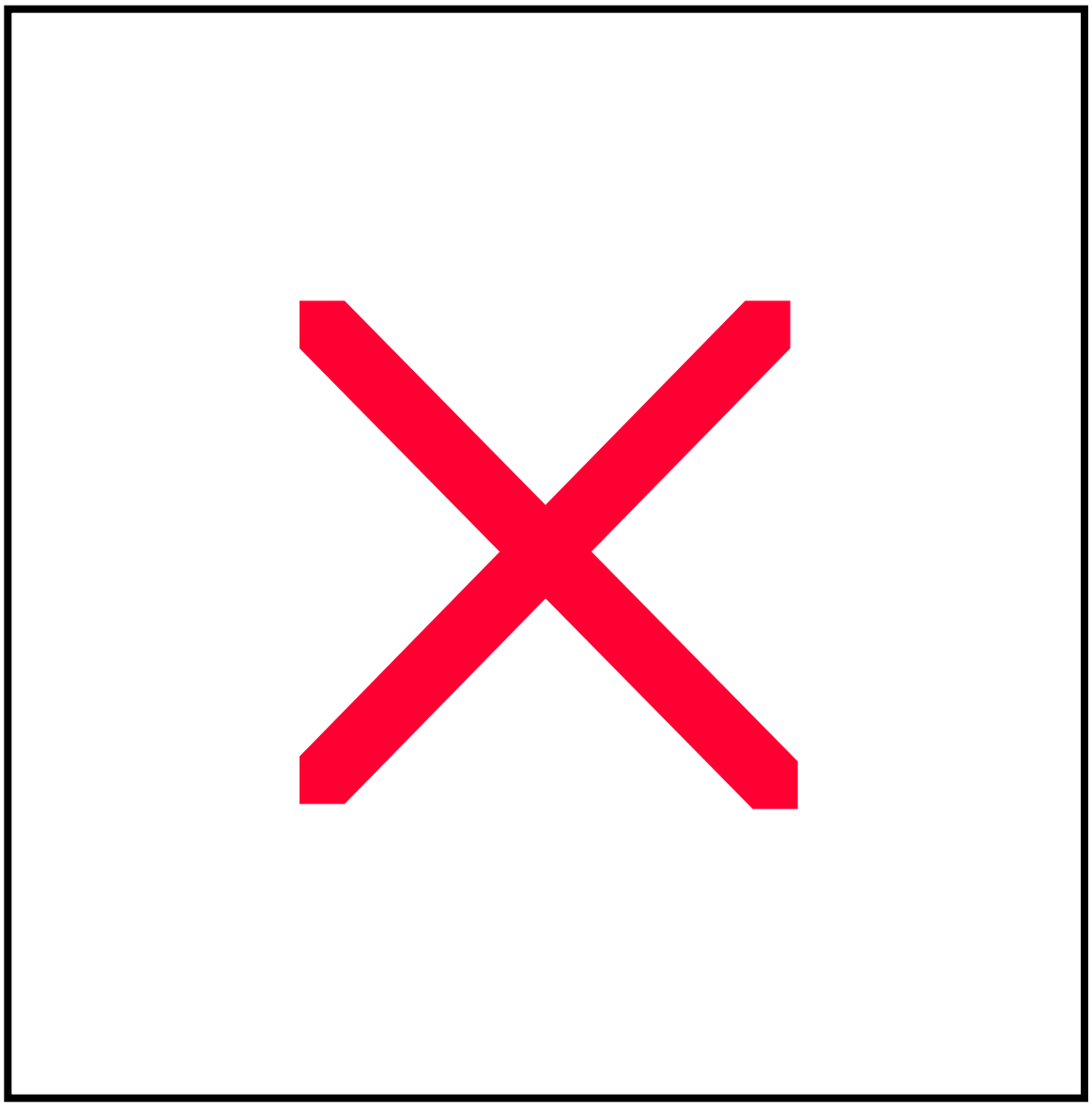

Figure 6
Actual Asphalt Content